10/807,872

What is claimed:

1. An aqueous antiperspirant active composition comprising admixing:

(a) a basic aluminum halide having the empirical formula

$$Al_2(OH)_{6-x1}\ Y_{x1}\ Rp\ (AA)q$$

wherein Y is Cl, Br, or I and $1.3 \leq x_1 \leq 1.7$, wherein R is an organic solvent having at least two carbon atoms and at least one hydroxy group and p has a value of $0 \leq p \leq 1.0$ and wherein (AA) is amino acid or amino acid compound and $0 \leq q \leq 0.5$ and aluminum material being further characterized by:

(i) size exclusion high performance liquid chromatography test band having a Band I relative area value of less than 5%, a Band II relative area value of 20% to 60%, Band III relative area value of 10% to 35% and Band IV relative area value of 15% to 50% and the sum of Band III and Band IV relative area value of at least 45% and no more than 70% and

(ii) ^{27}Al NMR spectrum wherein at least 45% of the total area under the spectrum from +100 ppm to -100 ppm is contained in the sum of the areas of resonance lines at or below 10 ppm and

(iii) in which the area of the resonance at 63 ppm is less than 0.1% of the total area under the spectrum from +100 ppm to -100 ppm and

(iv) which comprises 30 to 42% by weight of anhydrous basic aluminum halide antiperspirant active in water and

(b) at least one other antiperspirant active material which is selected from the group consisting of antiperspirant active of Zr salts, antiperspirant active of Hf salts, antiperspirant active of Ti salts and antiperspirant active of Sn salts.

2. The antiperspirant composition of claim 1 wherein nonaluminum antiperspirant compound is selected from the group having a following general empirical formula:

$$ZrO(OH)_{2-nz}B_z$$

wherein z may vary from 0.9 to 2, and n is the valence of B and 2-nz is greater than or equal to 0 and B is selected from the group consisting of halides and nitrate and with or without amino acid or salts of amino acids. As an alternative to or in conjunction with the above described zirconium salts, it is also possible to employ zirconium basic carbonate which has been represented by empirical formulas $[ZrO(OH)(CO_3)_{0.5} \cdot nH_2O]$ or $[Zr_2(OH)_4(CO_3)_2 \cdot nH_2O]$ which should not be interpreted as precise with respect to chemical structure but should be regarded only as a guide to molar ratio.

3. The antiperspirant composition of claims 1 wherein the amino acids can be glycine, DL-valine, alanine, arginine, lysine, and salts of amino acids can be sodium glycinate, calcium glycinate, magnesium glycinate, strontium glycinate and mixtures thereof.
4. The antiperspirant composition of claims 2 wherein the amino acids can be glycine, DL-valine, alanine, arginine, lysine, and salts of amino acids can be sodium glycinate, calcium glycinate, magnesium glycinate, strontium glycinate and mixtures thereof.
5. The antiperspirant composition of claim 2 wherein aluminum to zirconium molar ratio is from 1:10 to 10:1 and wherein the metal/halide molar ratio is 0.9:1 to 1.7:1.
6. The antiperspirant solution of claim 1 formed by mixing in an aqueous solution natural or synthetic antimicrobial compound.
7. The antiperspirant of claim 6 wherein the antimicrobial is selected from triclosan, triclocarbon, zinc compounds, green tea extract, Neem oil and mixtures thereof.
8. The antiperspirant solution of claim 1 where Y is chloride and Al:Cl molar ratio is 1.3:1 to 1.4 to 1.

9. The antiperspirant solution of claim 1 where Y is chloride and Al:Cl molar ratio is 1.2:1 to 1.5:1.

10. The antiperspirant solution of claim 1 where an amino acid is glycine.

11. The antiperspirant solution of claim 2 where an amino acid is glycine.

12. The antiperspirant solution of claim 2 containing an amino acid compound and wherein the amino acid compound is selected from an alkali metal, an alkaline earth metal, ammonium or hydroxy salt of an amino acid, a metal glycinate and a hydroxy aluminum salt of an amino acid.

13. The antiperspirant solution of claim 2 where amino acid compound is an alkali metal, an alkaline earth metal, ammonium or hydroxy salt of an aminoacid, a metal glycinate and a hydroxy aluminum salt of an amino acid.

14. The antiperspirant composition of claim 2 where amino acid salt is selected from sodium glycinate, magnesium glycinate, potassium glycinate, calcium glycinate, zinc glycinate and strontium glycinate and mixtures thereof.

15. The antiperspirant solution of claim 1 where the organic solvent is a polyhydric alcohol having at least three to about 12 carbon atoms and at least two hydroxy groups and is present at a concentration of about 1 to 10 weight percent.

16. The antiperspirant solution of claim 2 where the organic solvent is a polyhydric alcohol having at least three to about 12 carbon atoms and at least two hydroxy groups and is present at a concentration of about 1 to 10 weight percent.

17. The antiperspirant solution of claim 1 wherein the organic solvent is a polyhydric alcohol and is selected from glycerin, diglycerol, glyceridacid and mixtures thereof.

18. The antiperspirant solution of claim 2 wherein the organic solvent is a polyhydric alcohol and is selected from glycerin, diglycerol, glyceridacid and mixtures thereof.

19. The antiperspirant solution of claim 1 wherein the organic solvent is selected from the group consisting of ethylene glycol, polyethylene glycols, propylene glycol, dipropylene glycol, sorbitol, diethylene glycol, butylene glycol, hexylene glycol, 1,2-propylene glycol, 1,3 propylene glycol, glycerin, 1,2-hexanediol, hexanetriol, tripropylene glycol, propylene glycol methyl ether, isopropyl glycerol ether, dipropylene glycol methyl ether and combinations thereof at a concentration of about 1 to 10 weight percent.

20. The antiperspirant solution of claim 2 wherein the organic solvent is selected from the group consisting of ethylene glycol, polyethylene glycols, propylene glycol, dipropylene glycol, sorbitol, diethylene glycol, butylene glycol, hexylene glycol, 1,2-propylene glycol, 1,3 propylene glycol, glycerin, 1,2-hexanediol, hexanetriol, tripropylene glycol, propylene glycol methyl ether, isopropyl glycerol ether, dipropylene glycol methyl ether and combinations thereof at a concentration of about 1 to 10 weight percent.

21. The antiperspirant powder obtained by spray drying the solution of claim 1.

22. The antiperspirant powder obtained by spray drying the solution of claim 2.

23. The antiperspirant powder obtained by spray drying the solution of claim 6.

24. The antiperspirant powder of claim 21 which have an average particle size of about 15 to 30 microns.

25. The antiperspirant powder of claim 22 which have an average particle size of about 15 to 30 microns.

26. The antiperspirant powder of claim 23 which have an average particle size of about 15 to 30 microns.

27. The antiperspirant powder of claim 22 which has bulk density from about 0.5gm/cc to 2 gm/cc.

28. The antiperspirant powder of claim 22 which is micronized to have an average particle size of about 1 to 15 microns.

29. A method of preparing an antiperspirant active solution comprising an admixture of:

A material having the empirical formula

$$Al_2(OH)_{6-x1}\ Y_{x1}\ Rp\ (AA)q$$

wherein Y is Cl, Br, or I and $1.3 \leq x_1 \leq 1.7$, wherein R is an organic solvent having at least two carbon atoms and at least one hydroxy group and p has a value of $0 \leq p \leq 1.0$ and wherein (AA) is amino acid or amino acid compound and $0 \leq q \leq 0.5$ and aluminum material and having:

(i) a size exclusion high performance liquid chromatography test band having a Band I relative area value of less than 5%, a Band II relative area value of 20% to 60%, Band III relative area value of 10% to 35% and Band IV relative area value of 15% to 50% and the sum of Band III and Band IV relative area value of at least 45% and no more than 70%;

(ii) an ^{27}Al NMR spectrum wherein at least 45% of the total area under the spectrum from +100 ppm to -100 ppm is contained in the sum of the areas of resonance lines at or below 10 ppm and

(iii) in which the area of the resonance at 63 ppm is less than 0.1% of the total area under the spectrum from +100 ppm to -100 ppm and

at least one other antiperspirant active metal cation material selected from the group consisting of antiperspirant active of Zr salts, antiperspirant active of Hf salts, antiperspirant active of Ti salts and antiperspirant active of Sn salts comprising the steps of:

(a) making basic aluminum halide by reacting aluminum powder, aluminum halide and water at a temperature greater than 85°C but below the reflux temperature;

(b) maintaining this reaction until the reaction products having Al:halide ratio of 1.2 to 1.5 and solution solids concentration of about 30 to 42 weight percent on an anhydrous basis;

(c) cooling said reaction products to about room temperature;

(d) filtering and aging said reaction products at about room temperature from about 1 day to 6 months until the desired size exclusion chromatograph is obtained; and

(e) admixing said reaction products with an amino acid or amino acid compound and with said metal cation material.

30. The method of claim 29 in which the basic aluminum halide is obtained by taking a conventional basic aluminum halide solution having a suitable aluminum to halide ratio adjustable to an Al:halide ratio of the solution to about 1.2:1 to 1.5:1 and adding an appropriate amount of aged or unaged lower basicity aluminum halide solution or HX or $AlX_3 . 6H_2O$ solution thereof, wherein X can be Cl, Br or I, and heating the aluminum halide to about 50°C - 100°C for a period of about 10 minutes to about 6 hours and thereafter aging the resulting aluminum halide at room temperature until the desired chromatographic distribution of aluminum species is obtained.

31. A method of claim 29 comprising the steps of making the basic aluminum halide; mixing a zirconium salt selected from those having the general formula:

$$ZrO(OH)_{2-nz}B_z$$

wherein z may be from 0.9 to 2 and n is the valence of B and 2-nz is greater than or equal to a 0 and B is selected from the group consisting of halides and nitrate at a mixing temperature ranging from room temperature and including reflux temperature to 100°C.

32. The method of claim 29 wherein a zirconium basic carbonate which has been represented by empirical formulas $[ZrO(OH)CO_{3\ 0.5} \cdot nH_2O]$ or $[Zr_2(OH)_4(CO_3)_2 \cdot nH_2O]$ as admixed with said basic aluminum halide.

33. The method of claim 29 wherein the said method includes the addition of an organic solvent before, during or after aging.

34. The method of claim 30 wherein the said method includes the addition of an organic solvent before, during or after aging.

35. The method of claim 31 wherein the said method includes the addition of an organic solvent before, during or after the admixing of a zirconium salt with basic aluminum halide of invention.

36. The method of claim 29 wherein the basic aluminum halide antiperspirant active is first buffered with an amino acid and or a salt of amino acids followed by the addition of an organic solvent.

37. The method of claim 30 wherein the desired basic aluminum halide antiperspirant active is first buffered with an amino acid and or a salt of amino acids followed by the addition of an organic solvent.

38. The method of claim 31 wherein the desired basic aluminum halide antiperspirant active is first buffered with amino acid and or a salt of amino acids followed by the addition of an organic solvent.

39. The method of claim 31 wherein the reaction product obtained is spray dried to powder.

40. The method of claim 39 wherein the said method further comprises micronizing or screening or air classification or combination thereof to achieve the desired particle size distribution, particle shape distribution and density.

41. A method of spray drying basic aluminum halides and compositions as in claim 39 wherein the atomizer used is csc disc or two fluid nozzle or single fluid nozzle or multiple drilled hole disc or porous metal disc.

42. A method of claim 31 wherein the reaction product is dried using any known suitable conventional industrial method.

43. A method making aluminum zirconium antiperspirant as in claim 39 wherein the dried powder has a loss on drying when kept at 105°C for 2 hrs. from 5% to 20% by weight.

44. A method of making aluminum zirconium antiperspirant in claim 40 wherein the particles comprising thin walled or thick walled hollow spheres, solid spheres and irregular shaped non-hollow particles in an admixture suitable to achieve the desired particle size and shape distribution.

45. A method of making aluminum zirconium antiperspirant as in claim 43 wherein the critical humidity of the product is about 5%-20%.